PATENT ABSTRACTS OF JAPAN

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(71)Applicant : SUMITOMO CHEM CO LTD
(72)Inventor : KANEMITSU AKIYOSHI

(54) PRODUCTION OF POLYETHERSULFONE RESIN FILM

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain a polyethersulfone resin film improved in color tone, esp. in the degree of yellowing, by adding hypophosphorous acid and/or phosphorous acid to a polyethersulfone resin soln., then casting the soln. onto a carrier, and peeling the resultant film from the carrier.
SOLUTION: A polyethersulfone resin having repeating units represented by the formula: [-Ph-SO2-Ph-O-] (wherein Ph is phenyl) is dissolved in a solvent (e.g. N,N-dimethylformamide. N-methyl-2-pyrrolidone methylene chloride, or chloroform) to give a resin soln. having a viscosity of 15 P or lower. After 50-200 ppm hypophousphorous acid and/or phosphorous acid is added to the soln., it is cast on a carrier, dried by evaporating the solvent, and peeled. Thus is stably produced a polyethersulfone resin film which exhibits a low degree of yellowing, an excellent clarity, a good surface smoothness and a high thickness accuracy and is suitable as an optical film.

L1 ANSWER 1 OF 3 HCA COPYRIGHT 2009 ACS on STN
AN 130:169310 HCA Full-text
ED Entered STN: 20 Mar 1999
TI Manufacture of polyethersulfone films with good yellowing prevention
IN Kanemitsu, Akiyoshi
PA Sumitomo Chemical Co., Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 3 pp.
CODEN: JKXXAF
DT Patent
LA Japanese
IC ICM C08J0005-18
ICS B29C0041-12; B29C0041-24; C08K0003-32; C08L0081-06; B29K0071-00; B29L0007-00
CC 38-3 (Plastics Fabrication and Uses)
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI JP 11035705	A	19990209	JP 1997-199941	19970725 <--
PRAI JP 1997-199941		19970725		

CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
JP 11035705	ICM	C08J0005-18
	ICS	B29C0041-12; B29C0041-24; C08K0003-32; C08L0081-06; B29K0071-00; B29L0007-00
	IPCI	C08J0005-18 [ICM,6]; B29C0041-12 [ICS,6]; B29C0041-24 [ICS,6]; C08K0003-32 [ICS,6]; C08L0081-06 [ICS,6]; B29K0071-00 [ICS,6]; B29L0007-00 [ICS,6]

AB Title films, useful for optical films, are manufactured by casting polyethersulfones (PES) solns. containing hypophosphonic acid and/or phosphonic acid on substrates, drying the solns. to form films, and peeling the films off the substrates. Thus, a Cl2CH2 solution containing PES composed of 82.4 mol% C6H4SO2C6H4O and 17.6 mol% C6H4SO2C6H4OC6H4C6H4O and 100 ppm (on PES) hypophosphonic acid was cast on glass and dried to give a film showing total light transmittance 89.7%, haze 0.4%, and yellowness index 0.92.
ST polyethersulfone film hypophosphonic acid yellowing inhibitor; phosphonic acid polyethersulfone film yellowing inhibitor
IT Yellowing prevention
Yellowing prevention
(agents; manufacture of polyethersulfone films containing hypophosphonic acid and/or phosphonic acid for yellowing prevention)
IT Polysulfones, uses
Polysulfones, uses
RL: POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); USES (Uses)
(polyether-; manufacture of polyethersulfone films containing hypophosphonic acid and/or phosphonic acid for yellowing prevention)
IT Polyethers, uses
Polyethers, uses
RL: POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); USES (Uses)
(polysulfone-; manufacture of polyethersulfone films containing hypophosphonic acid and/or phosphonic acid for yellowing prevention)
IT Discoloration prevention agents
Discoloration prevention agents
(yellowing; manufacture of polyethersulfone films containing hypophosphonic acid and/or phosphonic acid for yellowing prevention)
IT 9084-41-7D, Poly(oxyphenylenesulfonylphenylene), polymers with polyethersulfones 144041-35-0D, polymers with polyethersulfones
RL: POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); USES (Uses)

(manufacture of polyethersulfone films containing hypophosphonic acid and/or phosphonic acid for yellowing prevention)

IT 13598-36-2, Phosphonic acid 20267-10-1, Hypophosphonic acid
RL: MOA (Modifier or additive use); USES (Uses)
(yellowing inhibitors; manufacture of polyethersulfone films containing hypophosphonic acid and/or phosphonic acid for yellowing prevention)

L2 ANSWER 1 OF 3 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN
AN 1999-186388 [16] WPIX Full-text
DNC C1999-054911 [16]
TI Polyether sulphone group resin film manufacture, for liquid crystal elements - involves adding hypophosphorous or phosphorous acid to polyether sulphone group resin solution and applying on support body to form film which is dried and exfoliated
DC A26; A89; L03
IN KANEMITSU A
PA (SUMO-C) SUMITOMO CHEM CO LTD
CYC 1
PI JP 11035705 A 19990209 (199916)* JA 3[0] <--
ADT JP 11035705 A JP 1997-199941 19970725
PRAI JP 1997-199941 19970725
IPCR B29C0041-00 [I,C]; B29C0041-12 [I,A]; B29C0041-12 [I,C]; B29C0041-24 [I,A]; B29K0071-00 [N,A]; B29L0007-00 [N,A]; C08J0005-18 [I,A]; C08J0005-18 [I,C]; C08K0003-00 [I,C]; C08K0003-32 [I,A]; C08L0081-00 [I,C]; C08L0081-06 [I,A]
AB JP 11035705 A UPAB: 20050704
NOVELTY - Hypophosphorous or phosphorous acid is added to a polyether sulphone group resin solution and applied on a support body. The film is dried and then exfoliated from the support body. About 50- 2000 ppm of hypophosphorous or phosphorous acid is added to the resin solution.
In an example, about 0.08 weight part of hypophosphorous acid was dissolved in 2 weight parts of methanol and added to 98 weight parts of methylene chloride solution (A). The solution (A) was added to 15 weight parts of PES, 17.6 mole% of which has structural unit of -ph-SO2-ph-O-ph-ph-O- and 82.4 mole% of which has a structural unit of formula -ph-SO2-ph-O-, such that hypophosphorous acid was set at a concentration of 100 ppm. Further, methylene chloride was added so that the total amount with solution (A) becomes 85 weight parts. The resin solution was applied on a glass Petri dish and kept at room temperature. Then, it was dried at 120 °C for 8 hours and 90 °C for 8 hours. The PES film obtained was tested for the degree of yellowing which showed a low value of 0.92.
USE - For optical films, as substrate for liquid crystal elements, etc..
ADVANTAGE - The film has low degree of yellowing and provides high colour tone when used in optical elements. ABDT JP11035705
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ADVANTAGE
The film has low degree of yellowing and provides high colour tone when used in optical elements.
NOVELTY
Hypophosphorous or phosphorous acid is added to a polyether sulphone group resin solution and applied on a support body. The film is dried and then exfoliated from the support body.
EXAMPLE
About 0.08 wt part of hypophosphorous acid was dissolved in 2 weight parts of methanol and added to 98 weight parts of methylene chloride solution (A). The solution (A) was added to 15 weight parts of PES, 17.6 mole% of which has structural unit of -Ph-SO2-Ph-O-Ph-Ph-O- and 82.4 mole% of which has a structural unit of formula -Ph-SO2-Ph-O-, such that hypophosphorous acid was set at a concentration of 100 ppm. Further, methylene chloride was added so that the total amount with solution (A) becomes 85 weight parts. The resin solution was applied on a glass Petri dish and kept at room temperature. Then, it was dried at 120 °C for 8 hours and 90 °C for 8 hours. The PES film obtained was tested for the degree of yellowing which showed a low value of 0.92.
INORGANIC CHEMISTRY
Preferred composition - About 50- 2000 ppm of hypophosphorous or phosphorous acid is added to the resin solution.
FS CPI
MC CPI: A05-J06; A11-B05D; A12-L03B; L03-G05B

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(71)出願人　000002093
住友化学工業株式会社
大阪府大阪市中央区北浜４丁目５番33号

(72)発明者　金光　昭佳
愛媛県新居浜市惣開町５番１号　住友化学工業株式会社内

(74)代理人　弁理士　久保山　隆　（外１名）

(54)【発明の名称】　ポリエーテルスルホン系樹脂フィルムの製造方法

(57)【要約】

【課題】　高い色調を要求される液晶素子用基板や位相差フィルムなどの光学フィルムに適した黄変度が低いポリエーテルスルホン系樹脂フィルムの製造方法を提供する。

【解決手段】　ポリエーテルスルホン系樹脂を溶媒に溶解した樹脂溶液を支持体上に流延し、乾燥後、支持体から剥離してポリエーテルスルホン系樹脂フィルムを製造する方法において、樹脂溶液に次亜燐酸および／または亜燐酸を添加して行うことを特徴とする。

【特許請求の範囲】
【請求項１】ポリエーテルスルホン系樹脂を溶媒に溶解した樹脂溶液を支持体上に流延し、乾燥後、支持体から剥離してポリエーテルスルホン系樹脂フィルムを製造する方法において、樹脂溶液に次亜燐酸および／または亜燐酸を添加して行うことを特徴とするポリエーテルスルホン系樹脂フィルムの製造方法。
【請求項２】次亜燐酸および／または亜燐酸の添加量がポリエーテルスルホン系樹脂に対して５０～２０００ｐｐｍである請求項１記載の製造方法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明はポリエーテルスルホン系樹脂（ＰＥＳ）フィルムを製造する方法に関する。詳しくはフィルム色調に優れたＰＥＳフィルムを安定的に製造する方法に関する。液晶素子用基板や位相差フィルムなどの光学フィルムに要求される色調に優れたフィルムの製造に適した方法に関する。
【０００２】
【従来の技術】従来から、フィルムは種々の方法で製造されている。中でも、光学用途のフィルムは、溶融押出法と溶剤キャスト法で製造される場合が多い。溶融押出法は溶剤キャスト法に比べて製造工程が簡単であるが、未溶融物や異物除去が困難であるため、得られるフィルムには、ダイラインなどによる表面平滑性の低下や熱による着色が起きやすく、また異物等を核とする表面欠陥を生じ易く、光学フィルムの製造には必ずしも向いていない。一方、溶剤キャスト法は、溶剤を使うこと、乾燥工程が必要であることなど工程上の問題はあるものの、表面平滑性、厚み精度が良好であり、樹脂溶液をろ過することによって容易に異物除去ができ、また熱による着色がないため、光学フィルムの製造には適した方法である。
【０００３】
【発明が解決しようとする課題】しかしながら、液晶素子用セル基板や、位相差フィルムとしてＰＥＳフィルムを用いた場合、ＰＥＳ樹脂そのものの着色に起因する着色により、必ずしも満足できる品質のＰＥＳフィルムは得られていない。本発明者はかかる事情に鑑み、ＰＥＳフィルムの着色を低減させ、透明性を向上させるべく鋭意検討した結果、樹脂溶液に次亜燐酸および／または亜燐酸を添加して行うことにより、得られるＰＥＳフィルムの色調、特に黄変度を改善できることを見出し、本発明に至った。
【０００４】
【課題を解決するための手段】すなわち本発明は、ポリエーテルスルホン系樹脂を溶媒に溶解した樹脂溶液を支持体上に流延し、乾燥後、支持体から剥離してポリエーテルスルホン系樹脂フィルムを製造する方法において、樹脂溶液に次亜燐酸および／または亜燐酸を添加して行うことを特徴とするポリエーテルスルホン系樹脂フィルムの製造方法である。
【０００５】
【発明の実施の形態】本発明で用いられるＰＥＳは、通常、下式　化１
【０００６】
【化１】［－ｐｈ－SO_2－ｐｈ－Ｏ－］
（式中、ｐｈはフェニル基を表す。）で示される繰り返し構造単位からなる樹脂であるが、強度、耐久性、樹脂溶液の安定性など、種々の物性改善のために、他の繰り返し構造単位を含む共重合体であってもよい。このような繰り返し構造単位としては、下式　化２
【０００７】
【化２】［－ｐｈ　SO_2　ｐｈ－Ｏ　Ａ　Ｏ－］
（式中、ｐｈはフェニル基を、Ａは次式　化３または化４を表す。）
【０００８】
【化３】［－（ｐｈ）ｎ－］
（式中、ｐｈはフェニル基を、ｎは１～４の整数を表す。）
【０００９】
【化４】［－ｐｈ－（Ｂ－ｐｈ）ｍ－］
（式中、ｐｈはフェニル基を、Ｂは炭素数１～３のアルキレン基、酸素原子または硫黄原子を、ｍは１～３の整数を表す。）
【００１０】本発明で用いられる溶媒は、ＰＥＳを溶解するものであれば特に制限されるものではなく、例えば、Ｎ，Ｎ－ジメチルホルムアミド、Ｎ－メチル－２－ピロリドン、塩化メチレン、クロロホルムなどが挙げられる。これらの溶媒は２種以上を混合して用いることができる。また、添加される次亜燐酸、亜燐酸の樹脂溶液への溶解性を上げるため、上記のようなＰＥＳを溶解しやすい溶媒と、アセトン、メタノールのような、単独ではＰＥＳを溶解しにくい溶媒との混合溶媒を用いることもできる。
【００１１】樹脂溶液中のＰＥＳ濃度は、均一で安定な溶液が得られ、安定した流延が得られる粘度が確保されれば特に限定されるものではないが、ダイ、ドクターナイフなどを用いる場合は、乾燥時に風紋やさざ波状の欠陥が生じないようにするために樹脂溶液の粘度が約１５ポイズ以上になるような濃度が好ましい。また、樹脂溶液をシャーレなどの容器に流延する場合、あるいはスピンコート法などで行う場合は、樹脂溶液の広がり易さを考え、比較的低粘度（約１５ポイズより低め）になるような濃度が好ましい。
【００１２】本発明で用いられる支持体としては、通常の溶剤キャスト法で用いられるものが用いられ、ガラス板、離型処理を施したＰＥＴフィルム、金属ベルト、回転ドラムなどの種々の材質、形態のものが挙げられる。
【００１３】ＰＥＳ溶液の流延方法は、通常の溶剤キャ

スト法で用いられる方法が用いられ、スピンコート法、ドクターナイフ法を用いる方法、ダイを用いる方法、回転ロールを用いる方法、コンマロールを用いる方法などが挙げられる。

【００１４】ＰＥＳフィルムの製造は、上記の粘度になるよう樹脂濃度を調整した樹脂溶液を支持体上に流延し、乾燥した後、フィルムを剥離して行われる。乾燥方法は、特に制限されるものではないが、樹脂の耐熱温度、溶媒の揮発性を考慮して設定された温度の熱風によって行うのが好ましい。

【００１５】本発明において樹脂溶液に添加する次亜燐酸および／または亜燐酸の量は、通常、５０～２０００ｐｐｍ程度である。少なすぎると効果が小さく、多すぎると得られるフィルムが脆くなるなど他の物性に影響がでるので好ましくない。

【００１６】次亜燐酸および／または亜燐酸の樹脂溶液への添加方法は、特に制限されるものではない。一般に極性の低い有機系溶媒には溶解されにくいので、樹脂の溶解性を損なわない範囲で、水、メタノールなどの比較的極性の高い溶剤を用い、混合溶媒としてから、次亜燐酸および／または亜燐酸を添加、溶解させるか、あるいは比較的極性の高い溶剤に添加、溶解し、これを主溶媒と混合させる方法で通常行われる。

【００１７】

【発明の効果】本発明の方法により、黄変度が低いＰＥＳフィルムを製造することができ、特に高い色調を要求される液晶素子用基板や位相差フィルムなどの光学フィルムを製造する方法として適している。

【００１８】

【実施例】以下、本発明を実施例で詳細に説明するが、本発明はこれら実施例に限定されるものではない。なお、フィルムの厚みはミツトヨ社製マイクロメータを用いて、全光線透過率およびヘイズは日本精密光学（株）社製ポイック積分球式ヘイズメーターを用いて、黄変度（ＹＩ）は日本電飾社製色彩色差計を用いて測定した。

【００１９】実施例１

次亜燐酸０．０８重量部をメタノール２重量部に溶解させ、これを塩化メチレン９８重両部と混合し、次亜燐酸の塩化メチレン溶液（Ａ）を得た。下式　化５で示される繰り返し構造単位を８２．４モル％、および化６で示される繰り返し構造単位１７．６モル％を有するＰＥＳ１５重量部に、ＰＥＳに対して次亜燐酸が１００ｐｐｍになるように溶液（Ａ）を加え、さらに溶液（Ａ）との合計量が８５重量部になるように塩化メチレンを加え、樹脂溶液を得た。

【００２０】

【化５】$[-ph-SO_2-ph-O-]$

【００２１】

【化６】$[-ph-SO_2-ph-O-ph-ph-O-]$

【００２２】この樹脂溶液をガラスシャーレに注液、流延させた後、室温にて一昼夜静置し、溶剤を揮発さた。ガラスシャーレから取り出した後、９０℃で８時間、１２０℃で８時間乾燥させ、ＰＥＳフィルムを得た。得られたフィルムの評価結果を表１に示す。

【００２３】実施例２～４、比較例１～８

表１に示す添加剤およびその量を用いた以外は実施例１と同様に行いＰＥＳフィルムを得た。得られたフィルムの評価結果を表１に示す。

【００２４】

【表１】

	添加剤	添加量 (ppm)	全光線透過率 (%)	ヘイズ (%)	黄変度 (YI)	フィルム厚み (μm)
実施例1	次亜燐酸	100	88.7	0.4	0.92	94
2	次亜燐酸	300	88.8	0.7	1.01	102
3	次亜燐酸	500	88.8	0.7	1.09	106
4	亜燐酸	300	88.0	0.6	1.20	103
比較例1	なし	0	88.6	0.8	1.43	90
2	なし	0	88.7	0.2	1.67	110
3	燐酸トリクレジル	300	88.0	1.1	1.38	101
4	燐酸トリ－ｍ－クレジル	300	88.6	1.1	1.49	98
5	燐酸トリフェニル	300	88.6	0.6	1.60	105
6	燐酸ジフェニル	300	88.6	0.8	1.63	104
7	亜燐酸トリフェニル	300	88.6	0.7	1.62	96
8	亜燐酸ジフェニル	300	88.6	0.6	1.48	87

フロントページの続き

(51)Int.Cl.[6]　　識別記号　　ＦＩ

// Ｂ２９Ｋ　71:00

　Ｂ２９Ｌ　7:00

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PRODUCTION OF POLYETHERSULFONE RESIN FILM

Bibliographic data | Mosaics | Original document | INPADOC legal status

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Inventor(s): KANEMITSU AKIYOSHI
Applicant(s): SUMITOMO CHEMICAL CO
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Abstract of **JP 11035705 (A)**

PROBLEM TO BE SOLVED: To obtain a polyethersulfone resin film improved in color tone, esp. in the degree of yellowing, by adding hypophosphorous acid and/or phosphorous acid to a polyethersulfone resin soln., then casting the soln. onto a carrier, and peeling the resultant film from the carrier. SOLUTION: A polyethersulfone resin having repeating units represented by the formula: [-Ph-SO2 -Ph-O-] (wherein Ph is phenyl) is dissolved in a solvent (e.g. N,N-dimethylformamide. N-methyl-2-pyrrolidone methylene chloride, or chloroform) to give a resin soln. having a viscosity of 15 P or lower. After 50-200 ppm hypophousphorous acid and/or phosphorous acid is added to the soln., it is cast on a carrier, dried by evaporating the solvent, and peeled.; Thus is stably produced a polyethersulfone resin film which exhibits a low degree of yellowing, an excellent clarity, a good surface smoothness and a high thickness accuracy and is suitable as an optical film.

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CLAIMS

[Claim(s)]
[Claim 1]In a method of casting a resin solution which dissolved polyether sulphone system resin in a solvent on a base material, exfoliating after desiccation and from a base material, and manufacturing a polyether sulphone system resin film, A manufacturing method of a polyether sulphone system resin film carrying out to a resin solution by adding hypophosphorous acid and/or phosphorous acid.
[Claim 2]The manufacturing method according to claim 1 whose addition of hypophosphorous acid and/or phosphorous acid is 50-2000 ppm to polyether sulphone system resin.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the method of manufacturing a polyether sulphone system resin (PES) film. It is related with the method of manufacturing stably the PES film which was excellent in the film color tone in detail. It is related with a method suitable for manufacture of the film excellent in the color tone required of optical films, such as a substrate for liquid crystal elements, and a phase difference film.
[0002]
[Description of the Prior Art]From the former, the film is manufactured by various methods. Especially, the film of an optical application is manufactured by the melting extrusion method and the solvent cast method in many cases. Although a melting extrusion method is simple for a manufacturing process compared with the solvent cast method, Since non-melt and tailing are difficult, on the film obtained, it is easy to produce the surface discontinuity which the fall of surface smoothness by a die line etc. and coloring by heat break out easily, and uses a foreign matter etc. as a core, and is not necessarily fit for manufacture of the optical film. On the other hand, although there are problems of a process -- that a solvent is used for the solvent cast method and a drying process is required -- surface smoothness and thickness accuracy are good, and since tailing can be easily done by filtering a resin solution and there is no coloring by heat, it is a method suitable for manufacture of an optical film.
[0003]
[Problem(s) to be Solved by the Invention]However, neither the cell substrate for liquid crystal elements nor the PES film of the quality which can not necessarily be satisfied by coloring resulting from coloring of PES resin itself when a PES film is used as a phase difference film is obtained. the color tone of the PES film obtained by carrying out to a resin solution by adding hypophosphorous acid and/or phosphorous acid as a result of inquiring wholeheartedly so that this invention person may reduce coloring of a PES film in view of this situation and it may raise transparency, especially yellowing -- it found out that a degree was improvable and resulted in this invention.
[0004]
[Means for Solving the Problem]Namely, in a method of this invention casting a resin

solution which dissolved polyether sulphone system resin in a solvent on a base material, exfoliating after desiccation and from a base material, and manufacturing a polyether sulphone system resin film, It is a manufacturing method of a polyether sulphone system resin film carrying out to a resin solution by adding hypophosphorous acid and/or phosphorous acid.

[0005]

[Embodiment of the Invention]PES used by this invention is usually a lower type. -izing 1

[0006]

[Formula 1][-ph-SO_2-ph-O-]

(ph expresses a phenyl group among a formula.) -- although it is resin which consists of a repeated structure unit shown, it may be a copolymer which includes other repeated structure units for various physical-properties improvements, such as intensity, endurance, and the stability of a resin solution. As such a repeated structure unit, it is a lower type. -izing 2 [0007]

[Formula 2][-ph-SO_2-ph-O-A-O-]

(ph expresses a phenyl group among a formula and A expresses the formation 3 of following formula , or ** 4.)

[0008]

[Formula 3][-(ph)n-]

(ph expresses a phenyl group among a formula and n expresses the integer of 1-4.)

[0009]

[Formula 4][-ph-(B-ph)m-]

(ph expresses a phenyl group among a formula, B expresses the alkylene group, oxygen atom, or sulfur atom of the carbon numbers 1-3, and m expresses the integer of 1-3.)

[0010]It is not restricted especially if the solvent used by this invention dissolves PES, and N.N-dimethylformamide, N-methyl-2-pyrrolidone, a methylene chloride, chloroform, etc. are mentioned, for example. These solvents can mix two or more sorts, and can be used. Since the solubility to the resin solution of the hypophosphorous acid added and phosphorous acid is raised, the mixed solvent of the solvent which is easy to dissolve the above PES(s), and a solvent like acetone and methanol which cannot dissolve PES easily if independent can also be used.

[0011]A solution uniform [the PES concentration in a resin solution] and stable is obtained, especially if the viscosity from which the stable flow casting is obtained is secured, are not limited, but when using a die, a doctor knife, etc., In order to keep the defect of a wind ripple or the shape of ripples from arising at the time of desiccation, the concentration that the viscosity of a resin solution will be about 15 poise or more is preferred. When casting a resin solution into containers, such as a petri dish, or when carrying out with a spin coat method etc., concentration which considers the ease of spreading of a resin solution and turns into hypoviscosity (lowering from about 15 poise) comparatively is preferred.

[0012]As a base material used by this invention, what is used by the usual solvent cast method is used, and various construction material, such as a PET film, a metal belt, and a rotating drum, and a thing of a gestalt which performed a glass plate and releasing treatment are mentioned.

[0013]A method for which a flow casting method of a PES solution is used by the usual solvent cast method is used, and a spin coat method, a method of using the doctor knife method, a method of using a die, a method of using a roll kneader, a method of using a comma roll, etc. are mentioned.

[0014]Manufacture of a PES film is performed by exfoliating a film, after casting a resin solution which adjusted resin concentration on a base material and drying so that it may become the above-mentioned viscosity. Although a drying method in particular is not restricted, it is preferred that a hot wind of heat-resistant temperature of resin and temperature

set up in consideration of the volatility of a solvent performs.
[0015]Quantity of hypophosphorous acid added to a resin solution in this invention and/or phosphorous acid is usually about 50-2000 ppm. When too small, an effect is small, and since other physical properties -- a film which will be obtained if too large becomes weak -- are affected, it is not desirable.
[0016]An addition method in particular to a resin solution of hypophosphorous acid and/or phosphorous acid is not restricted. Since it is generally hard to dissolve in a polar low organic system solvent, in the range which does not spoil the solubility of resin. After considering it as a mixed solvent using polar, comparatively high solvents, such as water and methanol, hypophosphorous acid and/or phosphorous acid are added and dissolved, or it adds and dissolves in a polar high solvent comparatively, and is usually carried out by a method of mixing this with a main solvent.
[0017]
[Effect of the Invention]the method of this invention -- yellowing -- a PES film with a low degree can be manufactured and it is suitable as a method of manufacturing optical films of which a high color tone is required especially, such as a substrate for liquid crystal elements, and a phase difference film.
[0018]
[Example]Hereafter, although an example explains this invention in detail, this invention is not limited to these examples. in addition -- in total light transmittance and Hayes, the thickness of a film uses Optical company make POIKKU integrating sphere type hazemeter Precision [Japanese] using the micrometer by Mitutoyo Corp. -- yellowing -- the degree (YI) was measured using the color difference meter by a Japanese electric-spectaculars company.
[0019]Example 1 hypophosphorous-acid 0.08 weight section was dissolved in the amount part of methanol duplexs, this was mixed with 98-fold methylene chloride Ryobe, and the methylene chloride solution (A) of hypophosphorous acid was obtained. Lower type To PES15 weight section which has 17.6 mol of repeated structure unit % shown by 82.4-mol % and ** 6 in the repeated structure unit shown by the-izing 5. The solution (A) was added so that hypophosphorous acid might be set to 100 ppm to PES, the methylene chloride was added so that the total quantity with a solution (A) might become 85 weight sections further, and the resin solution was obtained.
[0020]
[Formula 5][-ph-SO_2-ph-O-]
[0021]
[Formula 6][-ph-SO_2-ph-O-ph-ph-O-]
[0022]After making this resin solution pour in and cast into a glass petri dish, it settles at a room temperature one whole day and night, and it is volatilization **** about a solvent. After taking out from a glass petri dish, it was made to dry at 120 ** at 90 ** for 8 hours for 8 hours, and the PES film was obtained. The evaluation result of the obtained film is shown in Table 1.
[0023]Except having used the additive agent shown in the one to Examples 2-4 and comparative example 8 table 1, and its quantity, it carried out like Example 1 and the PES film was obtained. The evaluation result of the obtained film is shown in Table 1.
[0024]
[Table 1]

	添加剤	添加量 (ppm)	全光線透過率 (%)	ヘイズ (%)	黄変度 (YI)	フィルム厚み (μm)
実施例1	次亜燐酸	100	89.7	0.4	0.92	94
2	次亜燐酸	300	89.2	0.7	1.01	102
3	次亜燐酸	500	89.3	0.7	1.39	105
4	亜燐酸	300	89.0	0.5	1.20	103
比較例1	なし	0	89.6	0.8	1.49	90
2	なし	0	89.7	0.2	1.67	110
3	燐酸トリクレジル	300	89.0	1.1	1.88	101
4	燐酸トリ-n-ブチル	300	89.6	1.1	1.49	99
5	燐酸トリフェニル	300	89.6	0.6	1.60	107
6	燐酸ジフェニル	300	89.6	0.2	1.53	104
7	亜燐酸トリフェニル	300	89.6	0.7	1.62	95
8	亜燐酸ジフェニル	300	89.6	0.6	1.48	87